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**Some sources of error in measuring lattice parameters.** By CHESTER R. BERRY, *Research Laboratories, Eastman Kodak Company, Rochester 4, New York, U.S.A.*

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The accurate determination of lattice parameters requires consideration of certain sources of error which have been neglected until now. One factor is the radiation damage produced in the specimen by the measuring beam of X-rays. Although it is generally appreciated that color centers are produced in alkali halides and metal oxides by the radiation used in diffraction experiments, the corresponding changes in lattice parameters have been measured only in KCl (Berry, 1955). With KCl, an increase of lattice parameter of about 1 part in  $10^6$  was observed during exposure in the diffraction apparatus after about 15 minutes when using unannealed crystals, and after an hour with annealed crystals. Thus, a given specimen can be used only for very short times when high accuracy is required. These limitations do not apply to metals where displacement of atoms can occur only by direct collision of an atom with a photoelectron having an energy in excess of about 0.5 MeV. (Glen, 1955).

The well known photo decomposition of silver halides into metallic silver and halogen vapor has stimulated many workers to search for intermediate states of decomposition, associated with latent-image formation, which might be detected by observing changes in lattice parameters of photographic emulsion grains (Hess, 1943; Burgers & Mesritz, 1947; Brentano & Spencer, 1947; Berry, 1953; Jungmann & Staude, 1953; Waidelich, 1955). The results of such experiments have been quite inconsistent, some workers reporting changes in lattice parameters by irradiation as large as 2 parts in 1000 and others reporting only much smaller changes, if any. A possible reason for some of these contradictory results may be connected with another source of error in measuring lattice parameters. It has been shown (Berry, Van Horn & Griffith, 1954) that changes in lattice dimension as large as 2 parts in 1000 may arise from changing pressures exerted by the gelatin binder when a film is subjected to bending of the type which occurred in some of the moving-specimen arrangements used in much of the work cited here. Errors of a much smaller order may occur when specimens measured in vacuum are compared with those at atmospheric pressure, but even then differences of the order of 1 part in  $10^6$  may result.

In measurements of lattice parameters using electron diffraction, crystals are usually in a size range from a few tens to a few hundreds of Ångström units. In such small crystals, surface forces may alter the interatomic

spacings (Lennard-Jones & Dent, 1928). Although DuMond and his co-workers (DuMond & Bollman, 1936; Miller & DuMond, 1940) were unable to detect a definite parameter change in X-ray diffraction experiments with fine particles, decreases of 5 parts in 1000 were observed (Boswell, 1951) in electron diffraction from alkali-halide crystals having a size of about 30 Å. In measurements of this kind, it is necessary to consider the possibility of large deviations of the peak positions from the Bragg angle. On using the gas-scattering equation, as applied to small spherical crystals by Germer & White (1941), it was shown that the positions of the maxima may depend on the shape and size of the crystals in the specimen (Berry, 1952). Peak shifts of 2 parts in 100 may occur when the crystals are as small as about 25 Å, even in the absence of any surface forces.

#### References

- BERRY, C. R. (1952). *Phys. Rev.* **88**, 596.  
 BERRY, C. R. (1953). *Proc. R. P. S. Centenary Conf.*, London, 1951, p. 113.  
 BERRY, C. R. (1955). *Phys. Rev.* **98**, 934.  
 BERRY, C. R., VAN HORN, M. H. & GRIFFITH, R. L. (1954). *Acta Cryst.* **7**, 135.  
 BOSWELL, F. W. C. (1951). *Proc. Phys. Soc. A*, **64**, 465.  
 BRENTANO, J. C. M. & SPENCER, L. V. C. (1947). *Phys. Rev.* **71**, 142.  
 BRENTANO, J. C. M. & SPENCER, L. V. C. (1949). *J. Chem. Phys.* **17**, 944.  
 BURGERS, W. G. & MESRITZ, F. A. (1947). *Physica*, **13**, 490.  
 DUMOND, J. W. M. & BOLLMAN, V. L. (1936). *Phys. Rev.* **50**, 524.  
 GERMER, L. H. & WHITE, A. H. (1941). *Phys. Rev.* **60**, 447.  
 GLEN, J. W. (1955). *Advances in Phys.* **4**, 381.  
 HESS, B. (1943). *Phys. Z.* **44**, 245.  
 JUNGMANN, H. & STAUDE, H. (1953). *Z. wiss. Photogr.* **48**, 194.  
 LENNARD-JONES, J. E. & DENT, B. M. (1928). *Proc. Roy. Soc. A*, **21**, 247.  
 MILLER, P. H. & DUMOND, J. W. M. (1940). *Phys. Rev.* **57**, 198.  
 WAIDELICH, W. (1955). *Z. Phys.* **140**, 241.

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**Another graphical aid for the evaluation of absorption corrections by Albrecht's method.** By D. E. HENSHAW, *Department of Physics, University of Western Australia, Nedlands, Western Australia*

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Rogers & Moffett (1956) have described a graphical aid based on Albrecht's (1939) method, for evaluating absorption corrections to observed X-ray diffraction inten-

sities. It has become the practice in this Department to use a rather similar method, which may have advantages.

The reciprocal lattice of the crystal is plotted on a